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BECHER PROCESS FOR THE PRODUCTION OF SYNTHETIC RUTILE

NOMINATION FOR ENGINEERING HERITAGE RECOGNITION



PREPARED BY ENGINEERING HERITAGE WESTERN AUSTRALIA

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¹ Department of Mines, Industry Regulation and Safety, Mineral Sands,
<<https://www.dmp.wa.gov.au/Investors/Mineral-sands-1473.aspx>>, Government of Western Australia,
(Accessed 7 December 2020).

1. Introduction

Mineral sands are an important source of ore for zirconium, titanium, thorium, tungsten and other rare earth elements. Western Australia has significant deposits of mineral sands on the Swan coastal plain with mining having occurred, or continuing to occur, in a number of locations from Eneabba in the North to Beenyup (near Augusta) in the South.

After World War II, Australia was the largest producer of two of the main mineral sands' most valuable products, rutile and zircon. Rutile is a source material for titanium and zircon for zirconium. While the mineral sands' industry has been a relatively small part of Australia's mining industry compared with gold, coal, iron ore and other base metals, Australia has played an important role in the global supply of these raw materials for a number of end users. More information on Western Australia's mineral sands industry is provided in Appendix A.

While rutile and zircon can be separated by spirals in a wet plant or by magnets and electrostatic equipment in a dry plant, the most abundant mineral sand in Western Australia is ilmenite. Due to the inclusion of iron oxide, ilmenite is only about 56% titanium dioxide (TiO_2) and if Western Australia's mineral sands industry was to develop to its full potential, a sustainable method for removing the iron and increasing the concentration of TiO_2 in ilmenite was required.

Early plants for the processing of ilmenite were acid sulphate based plants with a waste that disposed of acidic waste into the ocean via sand dunes which suffered significant rust staining. A plant of this type operated for a time at Burnie in Tasmania in the late 1950s. In Western Australia the first and only plant of this type, which operated in the 1960s, was the Laporte plant in Australind. Laporte's iron rich wastes were transported in a pipeline on a wooden pier across Leschenault Inlet and into the sand dunes in the Leschenault Peninsula Conservation Park.

A chemist working for the WA Government, Robert (Bob) Becher, motivated by a wish to protect Western Australian beaches from rust staining by removing the iron in an alternative process, worked to overcome this problem. Bob Becher invented what became known as the Becher Process, a process for converting ilmenite to synthetic rutile which contains between 88 and 95% TiO_2 . This process increases the value of the product five-to-tenfold and has allowed Western Australia to become one of the world's major exporters of titanium dioxide.

Similar to other parts of the mining industry, there have been and are a large number of engineers working in the mineral sands industry. These engineers² developed the Becher Process into a commercial scale plant. To date a total of six synthetic rutile plants have been built in Western Australia, with two still in use, one at Capel and one at Muchea.

Recognising the importance of the Becher Process to engineers involved in the development of the mineral sands industry in Western Australia, Engineering Heritage Western Australia nominates 'The Becher Process for the Production of Synthetic Rutile' for an Engineering Heritage Marker.

² Minproc Engineers (Bob Wilde) had significant input in the TiWest JV developed in the early 1990s.

2. Nomination Letter

Learned Society Advisor
Engineering Heritage Australia
Engineers Australia
Engineering House
11 National Circuit
BARTON, ACT 2600

Name of work:

The Becher Process for the Production of Synthetic Rutile

This work is nominated for an Engineering Heritage Marker (EHM) under the Heritage Recognition Program of Engineers Australia.

Location:

The subject of this recommendation involved the invention, technological development and commercialisation of an innovative process for extracting a high-value product from mineral sands in Western Australia. As a process it has no specific location.

Owner:

While the process has been used by mineral sands companies, it has no owner. It was the subject of a patent awarded to Robert Becher, which has now expired. Accordingly, there is no requirement for an owner's agreement to the nomination. A leading user of the process, Iluka Resources, is aware of the nomination and has provided supporting information including images.

Access to site:

Not relevant.

Nominating Body:

Engineering Heritage Western Australia



Martin Silk
Chair
Engineering Heritage Western Australia
8 December 2020

3. Heritage Assessment

3.1 Basic Data

WA has long been recognised for its world-class resources and for having one of the most competitive and technologically advanced mining industries in the world. A significant part of this is the mineral sands industry, which includes the mining of ilmenite, an important source of titanium dioxide (TiO₂). TiO₂ is used in the production of paints, other coatings and plastics and a wide range of other applications.

Ilmenite has a TiO₂ content of about 56%, while synthetic rutile contains about 92%. Conversion of ilmenite to synthetic rutile increases the value of the product five- to tenfold, depending on market conditions. Commercial development of the process and the necessary technology was undertaken by mineral sands companies in conjunction with Dr Becher and his colleagues and Western Australia became one of the world's major exporters of synthetic rutile.

3.2 History

In January 1961 Dr Becher (Mr Becher at the time) lodged a Patent Specification (247110) covering the Becher Process for upgrading of ilmenite to synthetic rutile. The complete specification was accepted in September 1963. As the investigation work was done in a government laboratory the original patent was given to the West Australian Government. The process was commercialised in 1968 by the mineral sands company Western Titanium Ltd, a predecessor of Iluka Resources Ltd. The Becher Process has only been applied to ilmenite mined in Western Australia for direct manufacture of TiO₂ from synthetic rutile, based on the 'chloride' process.

3.3 Heritage Listings

There are no State Heritage listings for the Becher process. Curtin University have named Building 220A on the Perth Campus in Bentley the Becher building.

4. Assessment of Significance

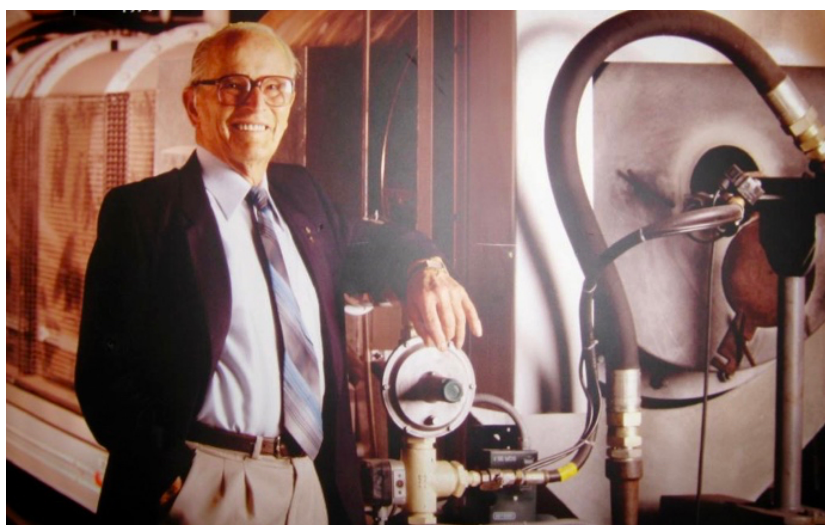
4.1 Historical Significance

The Becher Process is considered historically significant due to its invention and application at a time when Australia's production of minerals and mineral products were increasing after World War II. Its ability to significantly increase the value of mined ilmenite helped this trend and led to increased export income and jobs. It is also historically significant as an Australian invention that has enhanced the country's reputation for scientific and technological innovation.

4.2 Historic Individuals

The inventor of the Becher process was Robert Gordon Becher. He was born in 1913, moved with his family from South Australia to Perth in 1914 and left school at the age of 14. During

the Depression he worked at odd jobs. He went on to study at night school and completed a diploma in chemistry. He joined the Engineering Chemistry Division of the WA State Government Laboratories in 1952 and became heavily involved in minerals research.



Mr Bob Becher with equipment to undertake the Becher Process which is a breakthrough in the mineral sands industry. Image supplied by Department of Mines and Petroleum.

Dr Becher's substantial contribution to the mineral sands industry has been recognised in several ways. He received various awards and honours during his career, including a Fellowship of the Royal Australian Chemical Institute, the RACI Batty Medal in 1980, the Order of Australia in 1988, the Clunies Ross National Science and Technology Award in 1992 and an Honorary Doctor of Science Degree from Murdoch University in 1993. The naming of the R. G. Becher Building in 1996 on the campus of Curtin University took place on site and was attended by Dr Becher, then aged 82, and members of his family. He received several other honours for his achievements. He died in 1997.

In 2013 on the centenary of his birth the Bob Becher Centenary Symposium to celebrate his contributions to science and technology was held at the Australian Minerals Research Centre, Waterford WA.

4.3 Creative or Technical Achievement

As described by Jim Avraamides at the Centenary Symposium (Appendix D), Bob Becher's ideas for improving on the then current acid sulphate process were generated from the dumping of acidic iron waste into the ocean from a plant operating at Burnie in Tasmania in the late 1950s. With the building of a similar plant in Western Australia at Australind, he was keen to prevent this environmentally unacceptable situation being repeated in other locations in his home state.

In summary, the Becher process involves the reduction of the iron oxide in ilmenite to metallic iron in a kiln. This is followed by aeration or accelerated rusting. Accelerated rusting is achieved by mixing the reduced ilmenite in tanks with water and air, with ammonium chloride added to accelerate the process. The metallic iron dissolves, migrating through the pores, and precipitates as very fine iron oxide (0.1 to 10 microns in diameter).

The original grain loses up to 40% of its original mass, leaving a porous grain matrix which is largely titanium dioxide.

He devised a two-stage process which consisted of:

1. Reduction of the iron ore component in ilmenite at 1150° C with Collie coal char.
$$\text{FeO} \cdot \text{TiO}_2 = \text{Fe(m)} \cdot \text{TiO}_2$$

This was achieved using a modified iron reduction kiln.
2. Aeration leaching of the reduced ilmenite in an ammonium chloride solution to ‘rust out’ the iron and precipitate it as finely divided hydrated iron oxides.
$$\text{Fe(m)} \cdot \text{TiO}_2 = \text{Fe Oxides} + \text{TiO}_2$$



Mr Bob Becher's actual test unit that was used to develop the iron leaching step of the process. Image supplied by Iluka Resources.

The initial reduction tests were carried out using 6 grams of ilmenite and 6 grams of char. Ilmenite containing around 55% TiO₂ was readily upgraded to 90% or more TiO₂, while the iron oxide, which was almost a neutral pH by-product was readily contained and separated. A typical flow sheet is included as Appendix B.

Bob Becher applied for a Patent in 1961, using his own funds, which was granted in 1963. Western Titanium Ltd, a predecessor of Iluka Resources, commercialised the process in 1968 with the engineering development of the full-scale processing plant. A copy of the original patent specification is included as Appendix C.



*Aeration process at the Iluka Resources North Capel Operations.
Image supplied by Iluka Resources.*

Iluka Resources is WA's largest operator of the Becher Process. One of the company's predecessors was Western Titanium which saw in the Becher Process an opportunity to overcome the same problems with ilmenite upgrading as identified by Bob. The first 10,000 tonnes per annum (tpa) demonstration plant was constructed in Capel in 1969. In total 6 plants have been built using the Becher Process. Two are still in operation, one operated by Iluka in Capel and one operated by Tronox near Muchea. Local Collie coal has continued to be used as a fuel and as a reductant, so there was a natural synergy in the co-location of the processing plants in Western Australia.

The original capacity of the Tronox plant has increased from 130,000 tpa to 223,000 tpa. By 2013 total synthetic rutile production by Tronox had reached 4 million tonnes. By the same year Iluka had produced over 10 million tonnes of synthetic rutile by the Becher process.

4.4 Research Potential

The Becher Process was developed as a result of engineering/chemistry laboratory research. It has subsequently been commercialised and used as a basis for further research and development in the processing of mineral sands. The original process is still recognised by the industry and is likely to be the subject of ongoing development.

4.5 Social

The invention, commercialisation and further development of the Becher Process has assisted in the expansion of the Western Australian mineral sands industry, with increased jobs and economic benefits for the community.

4.6 Rarity

The Becher Process has only been applied to synthetic rutile manufactured from Western Australian Ilmenite for subsequent processing to titanium dioxide in the ‘chloride’ process. Other companies overseas may have adopted the Becher approach and developed further enhancements, but published information is limited.

4.7 Representativeness

The process is a good example of scientific research and technological development that has led to greatly increased product value. It is considered a good example of cooperation between government and private enterprise leading to economic benefits for industry and the community.

4.8 Statement of Significance

- The introduction of the Becher Process gave a significant boost to the mineral sands processing industry in Australia by inventing and developing a method for treating ilmenite to obtain synthetic rutile at a higher level of purity than other methods, while having lower environmental effects. In 2013 synthetic rutile made up around 20% of feedstock for global chloride pigment production.
- Development of the Becher Process is a good example of cooperation between government establishments and industry to produce a highly effective industrial process that has ongoing economic and social benefits.
- While the original process patent was awarded almost 60 years ago and various modifications have been made since, the Becher Process is still known as such and continues to be used.
- While Bob Becher’s career involved many other activities, he is still best known in Government and industry circles for his invention of the process and involvement in its development. The significance of this is demonstrated by the naming of the R. G. Becher building on the campus of Curtin University and the holding of the Bob Becher Centenary Symposium. A descriptive panel in the building and the symposium presentations both emphasise the Becher Process as central to his achievements and a reason for honouring his memory.

5. Interpretation Plan

A virtual interpretation is proposed for the Becher Process. The patent has long expired, so there is no recognised owner of the process. Its invention occurred when Dr Becher was working in the WA Government’s Chemical Laboratories in East Perth. These were renamed Chemistry Centre (WA), commonly known as ChemCentre, and in 2009 the organisation was relocated to new premises on the Curtin University campus at Bentley, a Perth suburb. Adjacent to this site is the CSIRO’s Waterford facility, which incorporates its Minerals Research Centre. This includes the Becher Building, which houses storage and laboratory

space. The building was named in 1996 when it was owned by the WA government and used by its Minerals Processing Laboratory. This was disbanded in the early 2000's and Dr Becher never worked there, so it really has little direct connection to him or the process. However it does contain a plaque inside the building commemorating the naming, and a panel containing information about him and the Becher Process. Any additional plaque and interpretation panel there would be superfluous.

The original East Perth site where the process was invented is no longer available or suitable for a physical recognition display. Another relevant point is that mineral sands companies, including RGC and Western Titanium and its successor Iluka Resources, took up the original Becher Process and carried out significant work to develop and commercialise it. It is also used by the company Tronox Resources. Enhancements to the process have continued to be made by the mining companies, CSIRO and others. It would be difficult to select a single representative location associated with the process.

Virtual recognition is therefore proposed. A “virtual interpretation panel” will be prepared for inclusion in the Heritage Database on the EHA web page.

6. References

1. Porter, Robert, *Below the Sands – The Companies that Formed Iluka Resources* (Consolidated Gold fields, RGC, Westralian Oil, Westralian Sands, Associated Minerals Consolidated, Western Titanium)
2. Notes from the Bob Becher Centenary Symposium: 26 November 2013
3. Notes on Iluka Resources from Wikipedia.
4. Mineral Sands – Technical Information, Iluka Resources.
5. Iluka's Synthetic Rutile Production June 2012
6. Government of Western Australia – Former Department of Commerce web pages:
<https://www.commerce.wa.gov.au/sites/default/files/atoms/files/8.3-developing-ip-case-study-3-published-version.pdf>
<https://www.commerce.wa.gov.au/sites/default/files/atoms/files/8.2-metallurist-who-cared-case-study-2-published-version.pdf>
<https://www.commerce.wa.gov.au/sites/default/files/atoms/files/8.1-ps-innovator-case-study-1-published-version.pdf>

7. Acknowledgements

The author wishes to thank the following for their assistance in providing information:

Nick Bernard, Technical Development Manager, Iluka Resources;

Robert Porter (for a copy of the “Below the Sands” book);

Members of Engineering Heritage Western Australia (EHWA) Committee for review of drafts and suggestions.

This nomination was prepared for EHWA by Mark Wanshaw.

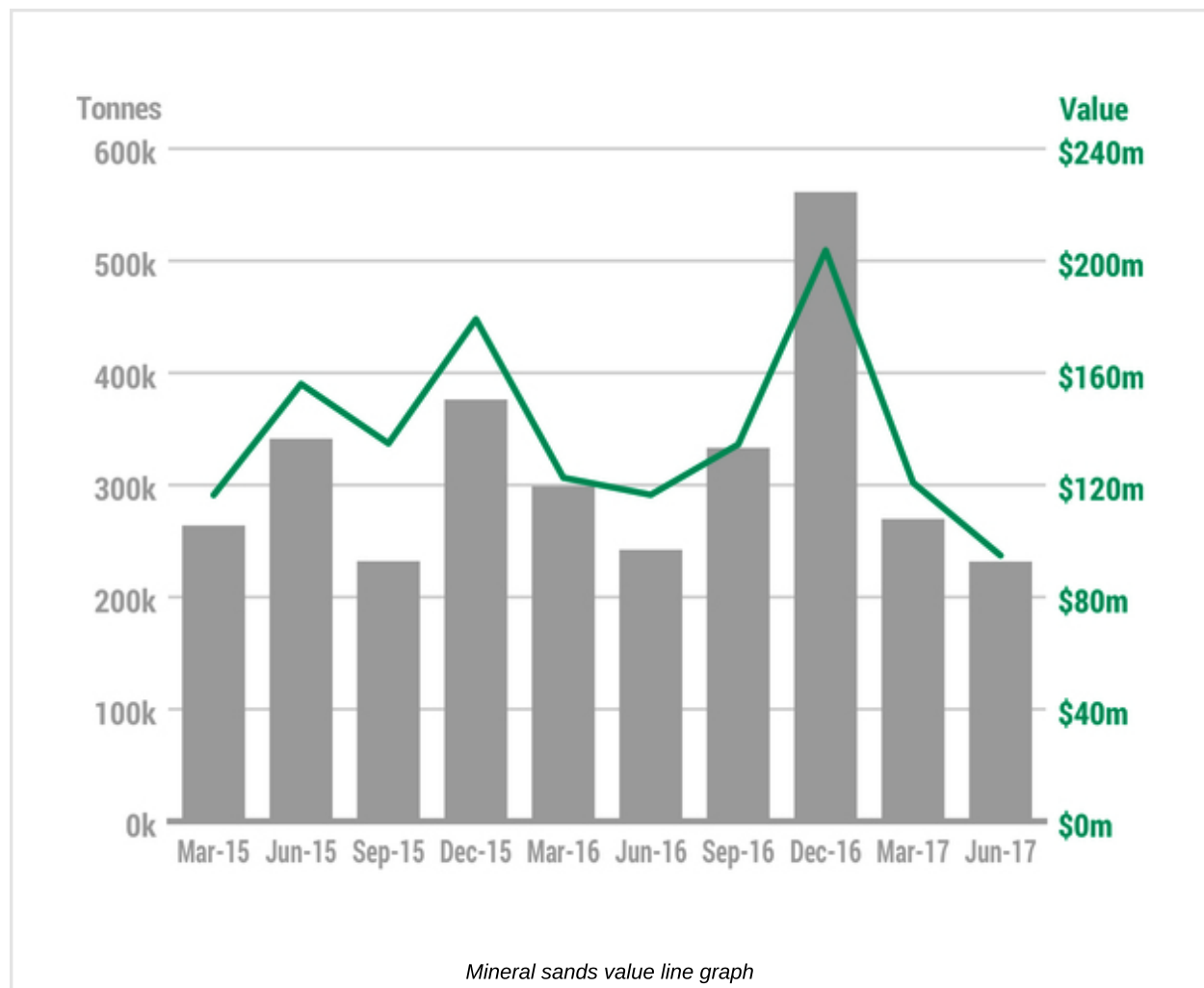
Appendix A : Mineral Sands in Western Australia



Government of **Western Australia**
Department of **Mines, Industry Regulation and Safety**

Mineral sands

A look at Western Australia



Australia is the world's leading supplier of mineral sands (ilmenite, rutile and zircon).

Mineral sands production in Western Australia predominantly comprises titanium minerals such as ilmenite, leucoxene, synthetic rutile and rutile. Other products such as garnet sand, zircon and staurolite are also produced on a smaller scale.

In 2016-17, Western Australia's mineral sands sector sold almost 1.4 million tonnes of material valued at around \$554 million.

Mineral sands production in Western Australia is dominated by two producers, Iluka Resources and Tronox Management, who produce the majority of the state's titanium minerals. GMA Garnet is another major producer, and currently the state's sole producer of industrial garnet sand.

Amazingly, Western Australia supplies mineral sands to 35 countries across every inhabited continent. China and the United Kingdom were Western Australia's major mineral sands export markets in 2016-17, accounting for 25 per cent and 12 per cent of total exports respectively. Highlighting the diversity of export markets for mineral sands products, other major export destinations included Saudi Arabia (11 per cent) the United States (11 per cent) and the Netherlands (7 per cent).

Uses

The titanium minerals – ilmenite, leucoxene and rutile – are primarily used as feed stock for the production of titanium dioxide pigment, with a small amount also used in titanium metal and fluxes for welding rods and wires. Titanium dioxide is the most widely used white pigment because of its non-toxicity, brightness and very high refractive index. It is an essential component of consumer products such as paint, plastics and paper.

Almost all titanium minerals are used as feedstock to produce titanium dioxide pigment used in products such as paints, paper and plastics. A small proportion is also used in titanium metal, predominantly for the aerospace industry. Zircon has a number of applications, principally in the manufacture of ceramic tiles and sanitary ware.

More information

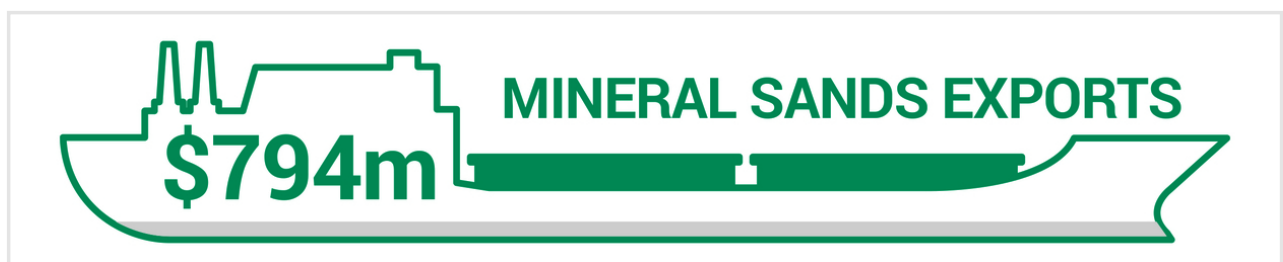
Australia is estimated to have the world's largest reserves of zircon with 48 million tonnes, that's around 64 per cent of the world's total estimated reserves of 75 million tonnes.

Australia is also estimated to have the largest rutile reserves at 27 million tonnes, which equates to 46 per cent of the world's total estimated reserves. Kenya and South Africa have the next largest reserves with 13 million tonnes (22 per cent) and 8.3 million tonnes (14 per cent) respectively.

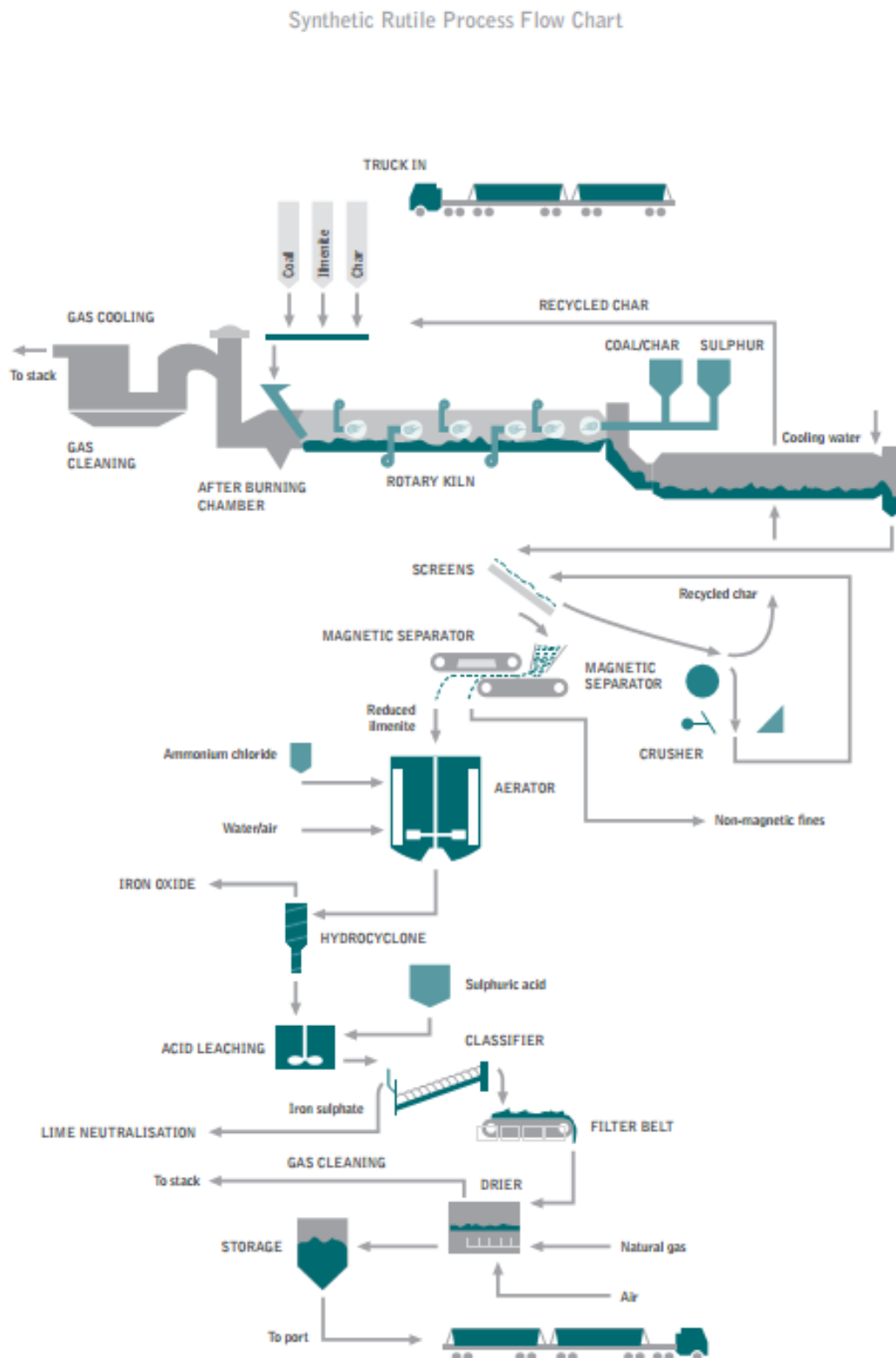
China is estimated to have the largest ilmenite reserves at 220 million tonnes. Australia ranks second with 150 million tonnes or 19 per cent of the world's total estimated reserves.

More detailed information about the performance of WA's mineral sands sector, and other commodities, can be found in the [Western Australian Mineral and Petroleum Statistics Digest \(/About-Us-Careers/Statistics-Digest-3962.aspx\)](#) or in the [latest resource data files \(/About-Us-Careers/Latest-Statistics-Release-4081.aspx\)](#).

Details about mineral sands mines, deposits, prospects and occurrences can be found in the Geological Survey of WA's [Titanium-Zircon Commodity Summary](#).
(<http://dmpbookshop.eruditetechnologies.com.au/product/titanium-zircon-heavy-mineral-sands-june-2013.do>).



Appendix B : Becher Process Flowsheet



Appendix C : Becher Process Patent Specification

247110

COMMONWEALTH OF AUSTRALIA PATENT SPECIFICATION

56, 550/60

Complete Specification Lodged 12th January, 1961.

Application Lodged (No. 56, 550/60) 15th January, 1960.

Applicant (Actual Inventor) Robert Gordon Becher.

Complete Specification Published 17th January, 1963.

Complete Specification Accepted 16th September, 1963.

Classification 14. 1; 14. 2; 14. 3; 15. 2.

International Classification C 22 b.

No drawing.

COMPLETE SPECIFICATION.

IMPROVED PROCESS FOR THE BENEFICIATION OF ORES CONTAINING CONTAMINATING IRON.

The following statement is a full description of this invention, including the best method of performing it known to me :-

This invention relates to an improved process for the beneficiation of ores containing contaminating iron. The process is particularly suitable for the beneficiation of iron contaminated ores such as ilmenite.

Iron is a constituent of many ores and must be removed during the treatment of the ore. It is well known that in producing titanium oxide pigments from ilmenite, one of the biggest problems is the elimination of the iron, which has a tendency to cause discolouration of the pigment. The treatment necessary to remove the iron greatly increases the cost of producing the pigments.

The object of the present invention is to provide a process for the removal of iron from ilmenite, and for the beneficiation of other iron contaminated ores or ferrites, which is relatively simple and inexpensive.

Broadly, the invention resides in a process for the beneficiation of ore containing contaminating iron which comprises roasting the ore with a reducing agent to reduce substantially all the iron present to the metallic state, separating the reduced ore from the gangue and unused reducing agent, agitating the separated ore under aerated water until the iron passes into solution and is precipitated as a light rust and separating the ore from the rust.

In one form the invention resides in a process for the beneficiation of ilmenite which comprises roasting the ilmenite with a reducing agent to reduce the iron oxides present

in the ilmenite to elemental iron, separating the reduced ore from the gangue and unused reducing agent, agitating the separated ore under aerated water until the iron passes into solution and is precipitated as a light rust and separating the ore from the light rust.

The reduction may be carried out by any known method in a rotary kiln or other suitable apparatus such as a fluid bed or saggars with a carboniferous reducing agent such as coke, coal, coal char or charcoal or with reducing gases such as hydrogen or gases derived from carboniferous materials.

Each particle of the reduced ore comprises an intimate mixture of elemental iron and oxides of the other elements present in the ore, and is separated from gangue and any unburnt reducing agent (when a solid reducing agent is used) by screening to separate the coarse particles and then a magnetic separation to separate the fines. Any other suitable method of separation may be used it being preferable to use a dry method for convenience of operation and handling of material. The reduced material freed from gangue and unburnt reducing agent is then placed in a container under water and air, oxygen or other oxidising gases blown through the water whilst the material is constantly agitated. The elemental iron present in the particles rapidly passes into solution and subsequently precipitates as a light precipitate of iron oxides which can be easily separated from the residual upgraded material by decantation or any suitable other conventional ore dressing procedure.

The rate of extraction of metallic iron from a reduced mineral by oxidation under water, hereinafter referred to as the oxidation of the iron, may be catalysed by the addition of a small amount of acid to adjust the pH to about four.

Other reagents which may be added to increase the rate of oxidation of the iron include oxidising agents, cations such as ammonium, the alkali elements, copper, or cobalt and organic anions such as acetates, chlorides, sulphates, nitrates fluorides and thionates. However, care must be taken in selecting the conditions for solution of the iron, otherwise some oxidation may take place in situ within the reduced ilmenite grain tending to regenerate "ilmenite" and impair the up-grading.

The rate of extraction or oxidation of the iron from the reduced mineral is also modified by the degree of agitation of the mineral in water and also the method of aeration employed. The agitation should be sufficient to keep the reduced ilmenite in suspension in the water. The aeration should be carried out in such a manner as to obtain a maximum solution of the oxidising gas. One method of doing this is by the use of any aeration apparatus giving a minimum size gas bubble using shear forces.

The oxidation may be carried out at atmospheric pressure and a temperature of 60-80°C. However, if required, the oxidation may be carried out under pressure - the exothermic heat of reaction of the oxidation of the iron being used to generate steam, which is then available for power production. When the reaction is carried out under pressure, the iron is oxidised at an increased rate, particularly in the presence of ferric ions which under these conditions (i.e. say 100 lbs/sq. in. and 150-170°C) are to a large extent regenerated.

If desired, the oxidation of the iron under water may be carried out using a mixture of air and carbon dioxide, in which case the resulting iron "oxide" is suitable for use in the absorption of sulphur gases.

Other gases which may be used in place of or in addition to air, oxygen and/or carbon dioxide include sulphur dioxide and nitrous oxide.

By controlling the conditions under which the oxidation is carried out various coloured iron oxides are obtained. For example cadmium and ferric ions promote the formation of yellow iron oxides while pretreatment of the reduced material with sodium carbonate

and a pH solution above five, promotes the formation of black iron oxide and a thixotropic mud may be used in this way. The colour of the iron oxides produced may also be varied by altering the composition of the aqueous media used, the bulk density of the system, the temperature at which the oxidation is carried out and the amount of available oxygen.

Whatever their mode of formation, the iron oxides produced in the process may be calcined at about 400 to 900°C to give different shades of red pigment. The oxides may also be reduced with hydrogen to give relatively pure iron powder.

The process described above has produced an upgraded ilmenite assaying up to about 90% TiO_2 from a raw material having a TiO_2 content of 56%.

The following examples illustrate the application of the process to the upgrading of ilmenite.

EXAMPLE 1 A 2000 gram sample of ilmenite assaying 56.6% TiO_2 , 18.2% Fe_2O_3 and 22.2% FeO was reduced carbon at 1000°C for 2 hours. On cooling, 1727 grams of reduced ilmenite was recovered by magnetic separation.

A 200 gram sample of this material was agitated and aerated in 1500 mls of water for 6 hours. The water contained in solution 1/2 gram of sodium sulphite and 1/2 gram of copper sulphate and had been adjusted to pH4 by addition of a few drops of H_2SO_4 .

On completion of aeration the rust produced was separated from the upgraded ilmenite by decantation and washing with water. 124 grams of upgraded ilmenite which assayed 90.0% TiO_2 and 5.0% FeO was obtained.

The iron oxides recovered weighed 122 grams and contained some upgraded ilmenite carried over in the decanting process. This material assayed 81.5% of iron oxides as Fe_2O_3 and 7.1% of TiO_2 . Generally these iron oxides contain about 10% of combined water.

EXAMPLE 2 Ilmenite assaying 56% TiO_2 and 40% $\text{FeO} + \text{Fe}_2\text{O}_3$ was reduced in a rotary kiln with carbon (coal) at a temperature of about 1150°C. The resulting reduced ilmenite assayed approximately 32% metallic iron. 43 lbs of this material was agitated and aerated under water for eight hours at a temperature of about 70°C. The water (40 gals) contained 20 mls of sulphuric acid and 300 grams of ammonium chloride; its pH at the beginning of the reaction was 4.3 and at the end of the reaction 5.3.

The progress of the reaction was measured by following the progressive decrease in reducing ions (ferrous ions) in solution by titration with 0.05N permanganated solution.

On completion of the reaction the iron oxides were separated by overflow pumping. The upgraded ilmenite was recovered from the reaction vessel and dried, 29 lbs being obtained assaying 89.3% TiO_2 .

A somewhat similar process is described in Australian Patent Specification No. 113, 553, wherein the elemental iron formed during the reduction step is oxidised in situ in the reduced ilmenite grain by air and moisture and the iron oxide then washed out of the grain. However, it has been found that there is a considerable mechanical barrier to the washing out of the iron oxide from the grain. The pores in the grain are formed in the reduction process by eating oxygen out of the ilmenite space lattice and it is difficult for the iron oxide molecule to be washed mechanically through the same hole. It is believed that the basis of the present process differs substantially from those described in Patent Specification No. 113, 553 in that by placing the reduced iron under water, the aerated water acts virtually as a dilute acid which dissolves the particles of iron but which does not attack the TiO_2 so that the particles of iron are removed in solution from the grain of reduced ore. Further aeration of the solution

brings about a precipitate of the iron oxide.

While the invention has been described with particular reference to the beneficiation of ilmenite, it is also applicable to the beneficiation of other ores or ferrites such as those of chromium, manganese, tantalum, zinc and nickel. It may also be applied to the treatment of oxidised and refractory gold ores, the reduction process producing elemental iron which can be oxidised under water to form iron oxides in a finely divided state so that the gold present is then amenable to cyanide attack.

The following example illustrates the application of the process to the treatment of refractory gold ore.

EXAMPLE 3 Calcined residues from a gold mine dump were sampled and assayed. The assay showed the residues to be about 50% iron oxides and to contain 5 dwts of gold per ton of residues. It was known that this gold was not soluble in cyanide and that it was mainly locked up in the iron oxide. The calcined residues were treated by the process disclosed in the patent in the following Manner.

100 grams of this material was mixed with coal char and heated to 950°C for 2 hours and on cooling, the reduced residues were recovered by screening and magnetic separation, 780 grams being obtained. This reduced material was then aerated under water and the rust so formed separated by decantation, 540 grams being obtained, leaving a residue of 340 grams.

The 540 gram lot, assayed 4 dwt to the ton and the residue 8 dwt to the ton. The cyanide soluble gold in the mixed product was 4 dwt to the ton. This experiment demonstrated that the above treatment of this refractory ore made the gold amenable to cyanidation.

The claims defining the invention are as follows:-

1. A process for the beneficiation of ore containing contaminating iron which comprises roasting the ore with a reducing agent to reduce substantially all the iron present to the metallic state, separating the reduced ore from the gangue and unused reducing agent, agitating the separated ore under aerated water until the iron passes into solution and is precipitated as a light rust and separating the ore from the rust. (The 15th January, 1960).
2. A process as claimed in Claim 1 wherein the precipitated ore component is separated by decantation or other conventional ore dressing procedures. (The 15th January, 1960).
3. A process for the beneficiation of ilmenite which comprises roasting the ilmenite with a reducing agent to reduce the iron oxides present in the ilmenite to elemental iron, separating the reduced ore from the gangue and unused reducing agent, agitating the separated ore under aerated water until the iron passes into solution and is precipitated as a light rust and separating the ore from the light rust. (The 15th January, 1960).
4. A process as claimed in any one of the preceding claims wherein the reducing agent is coal, coke, coal char or charcoal. (The 15th January, 1960).
5. A process as claimed in any one of the preceding claims wherein the rate of oxidation of the iron under the aerated water is increased by the addition of cations such as ammonium, the alkali metals, copper or cobalt and/or organic and inorganic anions such as acetates, chlorides, sulphates, nitrates, fluorides and thionates. (The 15th January, 1960).

6. A process as claimed in any one of the preceding claims wherein the pH of the water is adjusted to a PH of approximately 4. (15th January, 1960).

7. A process as claimed in any one of the preceding claims wherein the aeration of the water is carried out using a mixture of air and carbon dioxide. (15th January, 1960).

8. A process as claimed in any one of the preceding claims wherein the oxidation of the iron under the aerated water is carried out at atmospheric pressure and at a temperature of 60°-80°C. (15th January, 1960).

9. A process as claimed in any one of claims 1 to 8 wherein the oxidation is carried out under pressure. (15th January, 1960).

10. A process for the beneficiation of ores substantially as herein described. (15th January, 1960).

R. C. WRAY.

Patent Attorney for Applicant.

Related Art:

<u>Serial No.</u>	<u>Application No.</u>	<u>Classification.</u>
222, 517	22, 815/57	15. 2; 14. 2; 14. 1
"	7341/15	14. 2; 14. 3; 15. 2
126, 796	25, 394/45	15. 2; 14. 2.

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Appendix D : Report on Bob Becher Centenary Symposium

The Bob Becher Centenary Symposium: 26 November, 2013

Introduction

The year 2013 represents 100 years since the birth of Dr Bob Becher, Member of the Order of Australia and best known as the inventor of the Becher Process. This process, which is used by the West Australian mineral sands industry, is particularly suited to converting local weathered ilmenites into synthetic rutile feedstock for titanium dioxide pigment production.

A symposium to celebrate Bob's contributions to the minerals industry was held on 26 November, 2013 at the Australian Minerals Research Centre, Waterford in Western Australia. The Symposium's aim was to bring together those people who worked with, or have been influenced by Bob Becher in order to mark the centenary of his birth and to remember his contributions to science and technology. It was also fitting that the major sponsors of the symposium, Iluka Resources and TRONOX Resources, are users of Becher Process technology. The Royal Australian Chemical Institute, ChemCentre and CSIRO also provided support for the symposium. The AusIMM was represented by the Perth Branch president, Dr Chris Davis.

Background

Bob Becher was born in South Australia on 11 December, 1913 and he and his family moved to Perth in January, 1914. Bob received various awards and honours throughout his illustrious career, including a Fellowship of the Royal Australian Chemical Institute, the RACI Batty medal in 1980, Membership of the Order of Australia in 1988, the Clunies Ross National Science and Technology Award in 1992 and an Honorary Doctor of Science degree from Murdoch University in 1993.

He was recognised for his substantial contribution to minerals research in Western Australia when the State Government's Mineral Processing Laboratory facility (now part of CSIRO) at Waterford was named after him at its official opening in November, 1996.

Bob Becher died on 27 September 1997 aged 83. The Becher Process is his legacy - an example of his fine work as an exceptional scientist and innovator. Additionally, his career also included employment in the local brick making industry where he also made significant technical contributions.

The Symposium

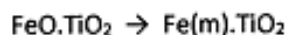
Attendance at the symposium was by invitation only and around 60 attended the half day event which was opened by WA's Chief Scientist, Professor Lyn Beazley. The Becher family was involved in the arrangements for the symposium and one of Bob's daughters and a granddaughter made short presentations on personal aspects of his life. Also in the audience were former colleagues of Bob who had been involved in the laboratory scale development of the process and the start-up of the first Becher Process demonstration plant in Capel in 1969. The technical program comprised six speakers from the WA government, CSIRO, the mineral sands Industry and academia. A short description of the presentations follows.

History of the Becher Process – Jim Avraamides (former Chief of the Mineral Processing Laboratory)

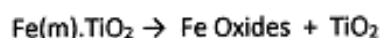
Bob joined the Engineering Chemistry Division of the WA State Government Chemical Laboratories in 1952. His ideas for improving on the then current acid sulfate process were

driven by his concerns on seeing the pollution generated from dumping of acidic iron wastes into the ocean from the plant operating at Burnie in Tasmania in the late 1950s. With the prospect of a similar plant being built in Western Australia, he was keen to prevent this environmentally unacceptable situation occurring in his home state. With a background in projects he had undertaken in production of sponge iron and making coke from Collie coal plus an inquiring and agile mind, he devised the 2-stage Becher Process which was patented (at his personal expense at the time) in 1963. Essentially, the two stages of the process are:

1. Reduction of the iron oxide component in ilmenite at 1150 °C with Collie coal char



2. Aeration leaching of the reduced ilmenite in an ammonium chloride solution to “rust out” the iron and precipitate it as finely divided hydrated iron oxides.



The initial reduction tests were carried out using 6 g of ilmenite and 6 g of char – a classic example of “from little things big things grow”. Ilmenite containing around 55 % TiO₂ was readily upgraded into a synthetic rutile of > 90 % TiO₂ while the (almost neutral pH) iron oxide by-product was readily controlled and contained. Western Titanium Ltd was supportive from the inception of the research and this collaboration extended to Bob and his colleagues assisting with the start-up of the first pilot plant constructed at Capel in 1968.

What the Becher Process meant to the world pigment industry and WA in particular – Nick Bernard (Technical Development Manager, Iluka Resources).

Iluka Resources is WA’s largest operator of the Becher Process. One of the company’s predecessors was Western Titanium Ltd which saw in the Becher Process an opportunity to overcome the same problems with ilmenite upgrading as identified by Bob. The first 10,000 tpa demonstration plant was constructed in Capel in 1969. Currently, Iluka operates 4 of the 5 reduction kilns having a total capacity of around 700,000 tpa of ilmenite. Originally, the Iluka kilns treated primary ilmenites which required a pre-oxidation stage but this was discontinued when weathered ilmenites (formerly a waste material) were found to respond well to Becher upgrading. Local Collie coal was ideal as a fuel and reductant so there was a natural synergy in the co-location of the ilmenite and coal deposits around the Capel area.

Synthetic rutile now makes up around 20 % of feedstock for global chloride pigment production. Iluka has so far produced over 10 Mt of synthetic rutile by the Becher Process.

The Becher Process at TRONOX – Andrew Struthers (Principal Engineer, TRONOX Resources).

TRONOX grew out of the original Cooljarloo JV and TiWest starting in 1988 with a mine located around 150 km to the north of Perth and a dry plant at Chandala near Muchea on the outer fringes of Perth. Synthetic rutile from Chandala is processed to pigment at TRONOX’s Kwinana operations. The Becher Process was chosen because:

- Cooljarloo ilmenites were compatible feedstocks with Collie coal;
- The process was already proven commercially and was in the public domain.

Because of the vertical integration of mine to upgrading plant to pigment plant, the TRONOX operation can factor in decisions taken at the mine and grades of synthetic rutile produced with planned outcomes at the pigment plant.

The original capacity of the reduction kiln has increased from 130,000 tpa to 223,000 tpa through having a good initial kiln design, high quality ilmenite feed, a program of debottlenecking and continuous improvement including better process control. By January, 2013, total synthetic rutile production had reached 4 Mt.

Understanding the Becher Process – Ian Grey (CSIRO Division of Process Science and Engineering).

In the early 1970s it was noted that the demonstration plant at Capel was not producing the expected grade of synthetic rutile. X-ray diffraction analysis of the reduced ilmenite from the kiln showed that the presence of impurities such as manganese and magnesium resulted in a separate phase being formed which contained unreduced iron i.e. $(\text{Mn,Fe,Ti})_3\text{O}_5$. A solution to the problem came from Peter Rolfe (WA Government Chemical Laboratories) in the form of adding sulfur to the kiln which segregated the manganese as MnS allowing iron oxide reduction to be improved. The MnS was removed by acid leaching and synthetic rutile grade improved from less than 90 % to 92 %. In commercial practice the sulfur was provided by using a waste product, copperas (iron sulfate), from a local sulfate pigment plant.

Amongst other work undertaken by Ian Grey was understanding of why weathered ilmenites are more amenable to kiln reduction than primary ilmenites. Primary ilmenites are denser so the reduction process is slower and iron tends to form on the surface of the ilmenite grains resulting in sintering. Pre-oxidation increases the porosity of the ilmenite grains thus enhancing reduction. Radioactive elements such as thorium and uranium posed another problem and CSIRO was asked to develop a removal procedure. This resulted in the SREP™ (Synthetic Rutile Enhancement) process where borate flux is added to the kiln charge. The radioactive elements are incorporated into the flux and, following aeration, are removed by acid leaching.

More work is continuing to develop and commercialise a NewGenSR™ process to treat primary ilmenites via pre-oxidation followed by hydrogen reduction and standard Becher aeration. A successful pilot plant trial was carried out in 2001-2003.

Rate Enhancements to the aeration stage of the Becher Process – Stuart Bailey (Curtin University, School of Applied Chemistry).

One of the first research projects to be carried out under the auspices of the AJ Parker Cooperative Research Centre for Hydrometallurgy was to re-examine the option of carrying out the aeration step under pressure and at higher temperatures. The aeration stage is relatively slow (12 – 24 hours) and Bob's early work and investigations by the industry confirmed increases in reaction rate of five-fold or more. The Parker Centre project also looked into developing a fundamental understanding of the aeration process, in particular the role of how ammonium chloride catalyst contributes to the removal of metallic iron from the reduced ilmenite and ensures it is deposited as iron oxide outside the reduced ilmenite particles.

The studies on the standard Becher aeration suggested that ammonium chloride's role was possible three-fold. Firstly, chloride is known to enhance corrosion of iron. Secondly, the presence of ammonia/ammonium ions assists in buffering the solution and inhibiting the precipitation of iron oxides within the reduced ilmenite grains. Thirdly, there is some evidence that ferrous ions (Fe^{2+}) form weak complexes with ammonia, allowing the dissolved iron to migrate from within the reduced ilmenite grain and into the bulk leach solution where it is oxidised further and precipitates as hydrated iron oxide.

High temperature leaching at above atmospheric pressure was tested on a number of reduced ilmenites using air or oxygen atmospheres. At 130 °C and 1 atmosphere pure oxygen pressure the rate of iron removal to a final level of 2-3 % was 7 times faster than for a standard atmospheric Becher aeration at 75 °C. Even greater rates were observed at higher temperatures and oxygen pressures but there was a trend to higher residual iron levels in the synthetic rutile product arising from in-situ deposition within the grains.

Bob Becher's Midland Brick R&D Career – (Tony Bagshaw, former Deputy Director, Chemistry Centre WA)

Following his time as a WA public servant, Bob joined Midland Brick in 1969 where he was employed until his retirement in 1979. His recollections of the time he joined were:

"When I did join them, I was employed as the research and quality manager with my background in chemistry. I didn't know much about bricks, but I did know a lot about science. Up until then Midland Brick wasn't exactly scientific. Brick making was pretty much as the Egyptians did it."

One of his tasks was to look at the problem of salts getting to the surface of bricks as they were being baked in the kilns. He latched onto using treacle in the mix and it worked! A patent was taken out for the use of sugar and this is now widely used by all brick makers. Another job was to investigate the emissions of fluorides from brick kilns which were located near wineries in the Swan Valley. This resulted in lime scrubbers being installed to capture these emissions. Bob was also proud of the new colours he developed for bricks and was always keen to point out "his bricks" to anyone who was with him when he was out and about.

Conclusions

During her opening address, Professor Lyn Beazley, WA's Chief Scientist, suggested that a Bob Becher Scholarship be established to encourage young researchers by offering them opportunities to work on a research project with an industrial application. This practical aspect would reflect Bob Becher's approach to using science for the community's benefit. The Symposium organising committee is preparing a draft proposal for circulation to interested parties.

Symposium organising committee:

Jim Avraamides, FAusIMM

John Farrow, FAusIMM

Tony Bagshaw, MAusIMM

Sarah Lau, MRACI